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LAMINATED FERROUS COMPOSITES BASED ON ULTRAHIGH CARBON
(UHC) STEELS (U) STANFORD UNIV CA DEPT OF MATERIALS
SCIENCE AND ENGINEERING O D SHERBY ET AL 30 JUN 87

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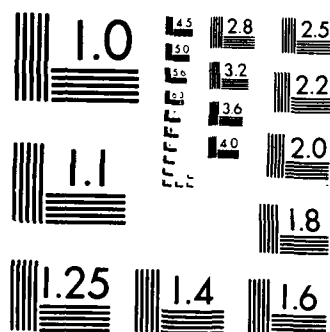
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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY DTIC		3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release; distribution unlimited. (2)	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE REFLECTED		4. PERFORMING ORGANIZATION REPORT NUMBER(S) 1 - 1507	
5a. NAME OF PERFORMING ORGANIZATION Stanford University		6b. OFFICE SYMBOL (if applicable) n.a.	
5c. ADDRESS (City, State, and ZIP Code) Department of Materials Science & Engr. Stanford, CA 94305		7a. NAME OF MONITORING ORGANIZATION U. S. Army Research Office	
7b. ADDRESS (City, State, and ZIP Code) P. O. Box 12211 Research Triangle Park, NC 27709-2211		8. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAG29-83-K-0153	
9a. NAME OF FUNDING / SPONSORING ORGANIZATION U. S. Army Research Office		9b. OFFICE SYMBOL (if applicable)	
9c. ADDRESS (City, State, and ZIP Code) P. O. Box 12211 Research Triangle Park, NC 27709-2211		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO.	PROJECT NO.
		TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) "Laminated Ferrous Composites Based on Ultrahigh Carbon (UHC) Steels" (Unclassified)			
12. PERSONAL AUTHOR(S) Oleg D. Sherby, Jeffrey Wadsworth, Dong Wha Kum, Glenn Daehn and Jerry Wittenauer			
13a. TYPE OF REPORT FINAL		13b. TIME COVERED FROM 1 Oct 83 TO 30 Apr 87	
		14. DATE OF REPORT (Year, Month, Day) 1987 June 30	
		15. PAGE COUNT 19	
16. SUPPLEMENTARY NOTATION The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
		Composites, laminates, mechanical properties, diffusion, superplasticity, fatigue strength, impact strength, ultrahigh carbon steel, stainless steel, Damascus steel	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) Ferrous laminated composites based on ultrahigh carbon (UHC) steel have been successfully manufactured by roll bonding below the A ₁ transition temperature. Three unique mechanical property characteristics have been achieved by such lamination. First, remarkably high notch-impact properties are achieved in such laminates. This is because notch-blunting occurs by delamination at the good (but not perfect) bond at the interface between laminates. Second, the fatigue properties of UHC steel are improved by lamination for the same reason. The requirements for delamination, however, are much more stringent for fatigue enhancement than for notch-impact enhancement. Third, non-superplastic materials, such as stainless steel, are made superplastic by lamination with fine-grained UHC steel. A ferritic stainless steel was shown to exhibit over 1000% elongation at 800°C when tested as a 15% stainless steel / UHC steel laminated composite.			
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL		22b. TELEPHONE (Include Area Code) 22c. OFFICE SYMBOL	

FINAL REPORTLAMINATED FERROUS COMPOSITES BASED ON
ULTRAHIGH CARBON (UHC) STEELS

(CONTRACT DAAG-29-83-K-0153)

Oleg D. Sherby, Jeffrey Wadsworth, Dong Wha Kum, Glenn Daehn and Jerry Wittenauer

This is the Final Report to the Army Research Office program entitled "Laminated Ferrous Composites Based on Ultrahigh Carbon (UHC) Steels". The program was monitored by Dr. George Mayer. The principal investigator was Oleg D. Sherby, Professor of Materials Science and Engineering, Stanford University, Stanford, California 94305. Dr. Jeffrey Wadsworth, Manager of the Metallurgy Department at the Lockheed Research Laboratories, Palo Alto, CA. contributed to the program as a Consulting Professor. The principal investigator and his colleagues (J. Wadsworth, D. W. Kum, G. Daehn and J. Wittenauer) are pleased to acknowledge Dr. Mayer's continuous guidance, interest and encouragement throughout all phases of this program. His help, advice and constructive comments were always sincerely appreciated.

Seven semi-annual progress reports were submitted during the life of this program. These progress reports summarize, in some detail, the various accomplishments made during the period 1 October 1983 to 31 December 1986. Formal meetings were held at least once a year at Stanford University on the progress of this research work. Visitors from a number of government research establishments, as well as from SRI International, were invited to, and attend these meetings. Specifically, meetings were held on 17 February 1984, 7 February 1985, 7 March 1986 and 17 October 1986. These meetings were sources of fruitful exchanges of information and ideas.

Three graduate students were supported during the course of the ARO Program. All three students are/were Ph.D. candidates and dissertations have or will be completed based on the ARO supported work. In the following sections, we summarize the work accomplished during the period of support from ARO through Contract DAAG-29-83-K-0153. The first section describes the students and their dissertation title, including their current and future status. The second section describes the publications which have resulted from the program. The third and last section summarizes the doctoral dissertation work of Kum, Wittenauer and Daehn.

I. DOCTORAL THESES BASED ON ARO PROGRAM

1. "Structure and Mechanical Behavior of Ferrous Laminated Composites Containing Superplastic Ultrahigh Carbon Steels". D. W. Kum

The thesis by Dr. Kum was completed in June 1984. Dr. Kum remained at Stanford as a post-doctoral fellow until his departure to Korea in March 1985. He is now a research scientist at the Korean Advanced Institute for Science and Technology (KAIST). Interaction by correspondence and by visits have led to continued association between Kum and Sherby and joint publications on work of mutual interest will be forthcoming.

2. "Factors Influencing the Fatigue Behavior of Ferrous-Based Laminated Composites". Jerry Wittenauer

The doctoral dissertation by J. Wittenauer is in the final stages of completion. It is anticipated that the thesis will be approved and submitted to the Graduate Division by August 1987. Mr. Wittenauer has accepted a post-doctoral appointment at the Research Laboratories of Sulzer Brothers in Winterthur, Switzerland. He will work with Drs. Bruno Walzer and H. Schlapfer.

3. "Superplasticity and Stability in Metal Based Composites".
Glenn Daehn

The doctoral dissertation by Glenn Daehn will be completed toward the end of October 1987. Mr. Daehn has accepted a position as Assistant Professor of Materials Science and Engineering at Ohio State University.

II. PUBLICATIONS BASED ON ARO PROGRAM

1. "Superplastic Behavior in Ferrous Laminated Composites", by B. C. Snyder, J. Wadsworth and O. D. Sherby, Acta Metall. 32, 1984, 919-932.

2. "Welded Damascus Steels and a New Breed of Laminated Composites", by J. Wadsworth, D. W. Kum and O. D. Sherby, Metal Progress, June 1986, 61-67.

3. "Development of Ferrous Laminated Composites with Unique Micro-Structures by Control of Carbon Diffusion", by D. W. Kum, T. Oyama, O. A. Ruano and O. D. Sherby, Metall. Trans. 17A, 1986, 1517-1521.

4. "Superplasticity of a Stainless Steel Clad Ultrahigh Carbon Steel", by Glenn Daehn, Dong Wha Kum and Oleg D. Sherby. Metall. Trans. 17A, 1986, 2295-2298.

5. "Factors Influencing the Fatigue Behavior of Ferrous Laminates", by Jerry Wittenauer and Oleg D. Sherby, J. Engr. Mat'ls. and Tech. 1987 (in press).

III. SUMMARY OF DISSERTATIONS BY D. W. KUM, J. WITTENAUER AND G. DAEHN

The three principal objectives of the ARO program were as follows:

- (1) develop novel processing methods for preparation of UHC steel/ferrous component laminated composites,
- (2) investigate the impact, fatigue and superplastic behavior of UHC steel laminated composites, and
- (3) evaluate the bond strength and bond structure of laminated composites as a function of processing conditions. Considerable progress was made toward achieving a number of goals within these broad objectives. A majority of our findings are given below by summarizing the dissertation work performed by D. W. Kum, J. Wittenauer and G. Daehn.

1. "Structure and Mechanical Behavior of Ferrous Laminated Composites Containing Superplastic Ultrahigh Carbon Steels" (D. W. Kum)

Ferrous laminated composites were manufactured by solid-state bonding techniques at temperatures below the A_1 , and discrete layers are developed in the composites. In this dissertation, stability of the discrete layers, superplastic behavior at 600-700°C, and impact properties at ambient temperatures of the composites were investigated. The results obtained are as follows:

- (1) The layer boundaries within the UHCS/mild steel laminated composite lose their discrete characteristics when the composite is subjected to severe deformation at temperatures just below the A_1 , or to extensive heat-treatment above the A_1 . It was shown that abnormal grain growth and diffusion (mainly carbon) are responsible for layer destabilization due to deformation and heat-treatment, respectively. The layers could be made stable, however, by increasing the activity of carbon through silicon addition in the low-carbon ferrous constituent. A UHCS/Fe-3%Si composite was developed and the layer boundaries in this composite were very stable. For example, laminated composite with extremely thin layers (2-5 μ m thick) with discrete boundaries could be made.
- (2) By tension testing parallel to the layers, total elongation of a UHCS/1005 laminated composite was 300-400% at 650-700°C, while that of the 1005 steel was only 120%. The high ductility in the composite is attributed to inhibition of void formation in the mild steel by diffuse necking and the presence of the superplastic UHCS layers.

- (3) Two analytical equations for creep in a direction perpendicular to the layers were developed. One is based on a uniaxial iso-stress model and the other on a biaxial isostrain model. Experimental results by compression testing of the UHCS/1005 laminated composite showed that the behavior could be predicted by a mixture of above two models, which is reasonable because specimens deformed in a manner wherein both isostress and iso-strain behavior can be expected.
- (4) Unusually high impact resistance was observed for a UHCS/1020 laminated composite and a UHCS/UHCS laminate when standard V-notch specimens were impact tested in the crack-arrestor orientation. The ductile-brittle transition temperature was -140°C and the upper shelf energy was greater than 325 Joules. This remarkably good behavior is due to notch blunting within the laminates as a result of delamination between layers. If the interlayer strength is improved by heat treatment, delamination does not take place and the impact properties are degraded.
- (5) A novel method has been developed for preparing ferrous laminated composites, containing ultrahigh carbon steel as one of the components, which results in hard and soft layers bounded by sharp and discrete interfaces. The method is based on increasing the activity of carbon in iron by silicon addition; in this manner, the carbon is made to segregate into specific layers by heat treatment at low temperatures (770°C). The results are ferrous laminated composites with discrete and sharp interfaces that consist of hard layers containing spherical carbide particles embedded in a matrix of ultrafine martensite or ferrite adjoining soft layers of a coarse grained iron alloy. In addition, the high activity of carbon is shown to result in total depletion of carbon in a silicon-containing UHC steel ribbon bonded to mild steel.
- (6) Laminated composites based on ultrahigh carbon steel/mild steel prepared in this investigation are similar to the welded Damascus steels of ancient times. The age-old saying "The best of the new is often the long forgotten past" may be applicable to the future use of ferrous laminated composites.

2. Factors Influencing the Fatigue Behavior of Ferrous-Based Laminated Composites". (Jerry Wittenauer)

The use of metallic-based laminates offers promise as a damage tolerant material under conditions of fatigue loading. It is known that laminated metals can exhibit improved impact properties due to a delamination and associated crack arrest phenomenon which has been observed to occur at weak interlaminar boundaries. In order to extend this crack arrest behavior to conditions of fatigue loading, a broad based experimental program has been conducted with ultrahigh carbon steel used as a base material.

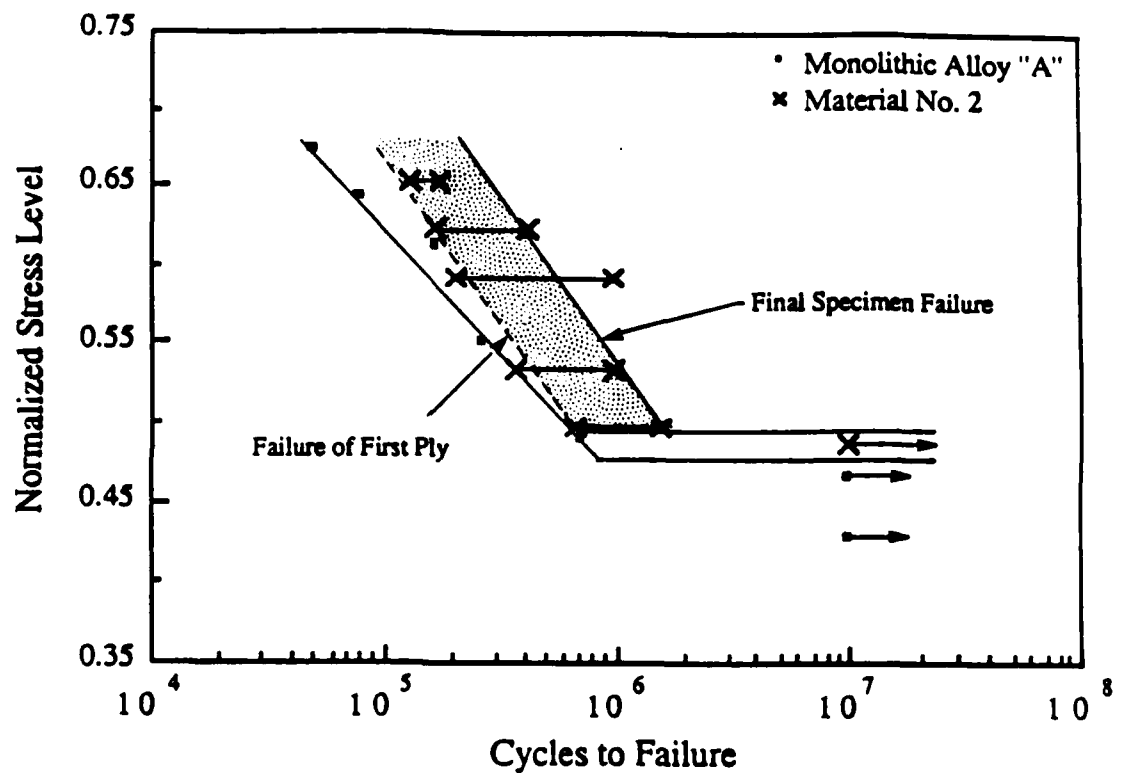
In the first phase of the experimental program, a shear test was used to evaluate factors influencing the interlaminar bond strength between adjacent steel plies. Since interlaminar bonds must be sufficiently weak for delamination and crack arrest to occur under fatigue conditions, a knowledge of interlaminar bond strength is critical if crack arrest mechanisms are to be optimized. A series of two-layer plates were mechanically roll bonded under various conditions of rolling reduction, rolling temperature, and post-bonding heat treatment. Using the shear test, it was found that the bond strength among steel plies remains quite high over a wide range of thermomechanical processing routes. A desire to obtain low interlaminar bond strengths led to the introduction of secondary metals to be used as weak interleafs between the high strength ferrous plies. Two materials - copper and iron containing three percent silicon - were evaluated and found to be suitable interleaf candidates. Interleaf selection criteria include reasonably high melting temperature, low yield strength, and minimal diffusivity with respect to the UHC steel. The use of copper as an interleaf results in an interlaminar bond which is about one-third as strong as the ultrahigh carbon steel. The use of the iron-silicon alloy as an interleaf produces an interlaminar bond which is one-half as strong as the ultrahigh carbon steel.

In the second phase of the experimental program, a variety of laminates consisting of layers of ultrahigh carbon steel interleaved with copper or iron-silicon alloy were prepared. The chemical composition of the steel used was 1.25% C, 0.49% Mn, .002% P, .006% S, 0.52% Si, and 1.52% Cr. The method of laminate preparation involved welding a stack of metal plates at the edges via the TIG weld process followed by high temperature mechanical roll bonding. Interleaf materials were inserted as thin foils between the sheets of ultrahigh carbon steel prior to welding. Typical rolling reduction used was ten to one resulting in a final thickness of 3 mm and all materials were rolled at 700° C. Ply thickness for the laminates studied ranged from 150 μ m to 1000 μ m and interleaf thickness ranged from 4 μ m to 50 μ m. The mechanical properties of the laminates were typically as follows: Yield Strength = 750 MPa, Ultimate Tensile Strength = 800 MPa, and elongation to failure of 20%.

Fatigue specimens were prepared from the roll-bonded sheet and tested under conditions of fully reversed bending fatigue. The fatigue behavior of laminates tested in the crack arrest orientation showed that a range of crack growth behavior is possible - ranging from crack arrest at each interleaf to uninhibited crack propagation. For laminates containing a copper interleaf, it was shown that crack arrest and life improvement are optimized when ply thickness is increased, when interleaf thickness is decreased, or when ferrous ply yield strength is increased. No life improvement was obtained through use of the ferrous interleaf. The fatigue behavior for the best material obtained is shown in the figure below. A life improvement by a factor of four is observed for the laminate when compared to a monolithic material of similar processing history.

A micromechanical model has been introduced which explains the experimentally observed crack growth behavior. By seeking an understanding of near crack-tip stresses as a fatigue crack propagates toward a weak interface, factors influencing local delamination and crack arrest can be fundamentally understood. A previously developed applied mechanics model was used for this purpose. The model used supports the experimental

results and has emerged as a potentially useful tool for predicting crack growth behavior in other material systems.



Stress-Life data for 9 ply laminate with 4 μm copper interleaf. The fatigue life for the laminate was improved due to crack arrest at the copper interleaf. All data is presented as being normalized to the UTS of the specimens. For the laminate, data is shown both for failure of the initial (outer) ply and for final specimen failure.

3. "Superplasticity and Stability of Laminated Composites Based on Stainless and Ultrahigh Carbon Steels". (Glenn Daehn)

I. INTRODUCTION

It is well known that single phase materials cannot be made inherently superplastic, since a second phase is necessary to stabilize an ultrafine grained structure at elevated temperature. This is unfortunate since there are many instances in which single phase materials are desired, and it may be advantageous to superplastically form such a material. The primary goal of the present work was to investigate the feasibility of producing laminated composites based on stainless steel roll bonded to the surface of a superplastic Ultrahigh Carbon Steel (UHCS). Previous work has shown that under proper conditions, laminated composites based on UHCS will behave superplastically. Such a procedure if applied to stainless steel results in a superplastic laminated composite, which, however, has the corrosion resistance of a single phase stainless steel.

In the ideal case, such a laminated composite should have many useful attributes. The surface would have the corrosion resistance of the stainless steel. The room temperature mechanical properties of the laminated composite may be vastly improved over those of the monolithic stainless steel due to the high strength of the core material. If a small volume fraction of stainless steel is used, strategic materials can be conserved. And, of course, the sheet can have the formability of a superplastic material, allowing intricate shapes to be readily and economically formed.

This report highlights developments in the study of such composite materials, and refinements to the concept presented above. This report will outline advancements in: the processing of such materials; their stability at elevated

temperature; the prediction of superplastic forming parameters; and, their gas pressure forming characteristics. The work presented shows that systems of this type are attractive candidates for further development and eventual use.

II. MATERIALS AND PROCESSING

Three-layer laminates consisting of about 15 volume percent stainless steel solid state bonded to a central core of UHC steel were examined in this study. Barrier layers were placed between the UHC and stainless steels in several of the laminates produced. The UHC steels examined had compositions as listed in Table 1.

Table 1. Compositions of UHC steels used in superplastic laminates

Designation	C	Cr	Mn	Si	Al
UHCS-3Si	1.26	1.50	0.54	2.98	0
UHCS-1.6Al	1.28	1.53	0.59	0.20	1.63

The function of the silicon and aluminum in the UHC steels is to stabilize the ferrite phase, raising the A_1 transformation temperatures in these materials. The aluminum also serves to further refine the grain structure.

Both a ferritic (26Cr-1Mo) and an austenitic (type 304) stainless steel were studied as cladding materials. In the range of temperatures and strain rates investigated for superplasticity, the austenitic stainless steel has a flow stress approximately 10 times that of the ferritic stainless steel. The compositions of these materials are listed in Table 2.

Table 2. Compositions of stainless steels used in superplastic laminates

Designation	Cr	Mo	Ni	C	Mn	Si	N	O
26Cr-1Mo	25.5	1.17	0.10	.001	n/a	0.22	.076	.0038
304	18.24	n/a	8.22	.031	1.63	0.4	n/a	n/a

Various barrier materials were studied for their ability to stabilize these composites against interdiffusion. The materials studied were: Oxygen Free High Conductivity (OFHC) copper (Cu), an alloy of iron and 3.2% aluminum (Fe-3.2Al), and interstitial free iron (I. F. Iron).

The production of laminated composites from these base materials proceeded as follows. The component materials are ground to a flat 400 grit finish and degreased. The plates are stacked and welded into a packet as shown in Figure 1.

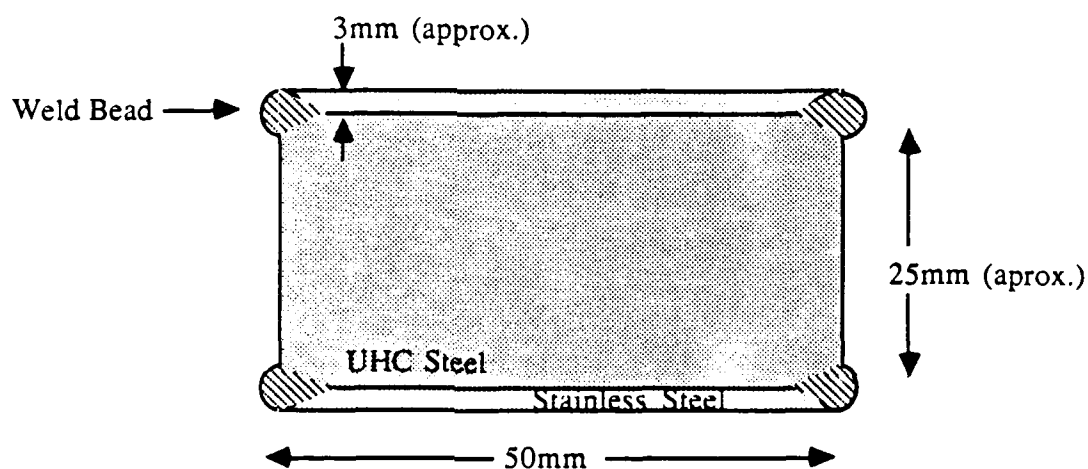


Figure 1. Schematic representation of the initial step in preparation of laminated composites used in superplasticity testing.

This packet is then given a normal DETWAD rolling procedure (i.e., heated slightly above the A_1 transformation temperature for about 30 min and rolled to an approximate 10:1 reduction while cooling). This procedure has two effects: it refines the UHC structure (about $2\mu\text{m}$ ferrite grain size), and it bonds the component materials together.

The three-layer laminates produced for use in this study are described in Table 3. Column by column, the table lists: the volume fraction of the stainless steel and barrier layers making up the composite, the final thickness of each composite, the final thickness of barrier layers, the reduction ratio in processing, and the soaking temperature prior to DETWAD rolling.

Table 3. Summary of Laminates Produced for Superplasticity Testing.

Laminate	Vol % non Superplas. compon's	Laminate thickness (mm)	Barrier thickness (μ m)	Roll Bond Reduction (ratio:1)	Soaking Temp. (deg C)	Notes
26Cr-1Mo/ UHCS-3Si	12.4	2.46	n/a	12.7	850	
26Cr-1Mo/ Fe-3.2Al/ UHCS-1.6Al	13.4	3.33	73.7	11.5	815	Fe-3.2Al and 26Cr-1Mo first bonded at 1100°C
26Cr-1Mo/ I.F. Iron/ UHCS-1.6Al	17.1	1.96	43.2	18.8	n/a	First HWW bond then DETWAD from 830° C
26Cr-1Mo/ Cu/ UHCS-1.6Al	16.0	3.12	13.0	9.75	810	Cu foil insert
304/ UHCS-1.6Al	16.4	2.44	n/a	16.7	815	
304/ Cu/ UHCS-1.6Al	15.2	2.13	6.6	19.0	815	Cu foil insert

DETWAD bonding can create laminates with very discrete interfaces. This is due to the relatively low bonding temperatures and short times available for interdiffusion. Figure 2 shows a typical DETWAD bonding interface. The commercial production of similar materials, such as stainless clad-structural steel, usually relies on high temperatures (about 1300°C), and large deformation to obtain effective bonding. A stainless steel / structural steel interface produced commercially by Kawasaki Steel is shown in Figure 3. The figure shows deep penetration of carbides into the stainless steel, as evidenced by the etching contrast. It is well known that carbon will destroy the corrosion-resisting properties of stainless steel by forming carbides with chromium.

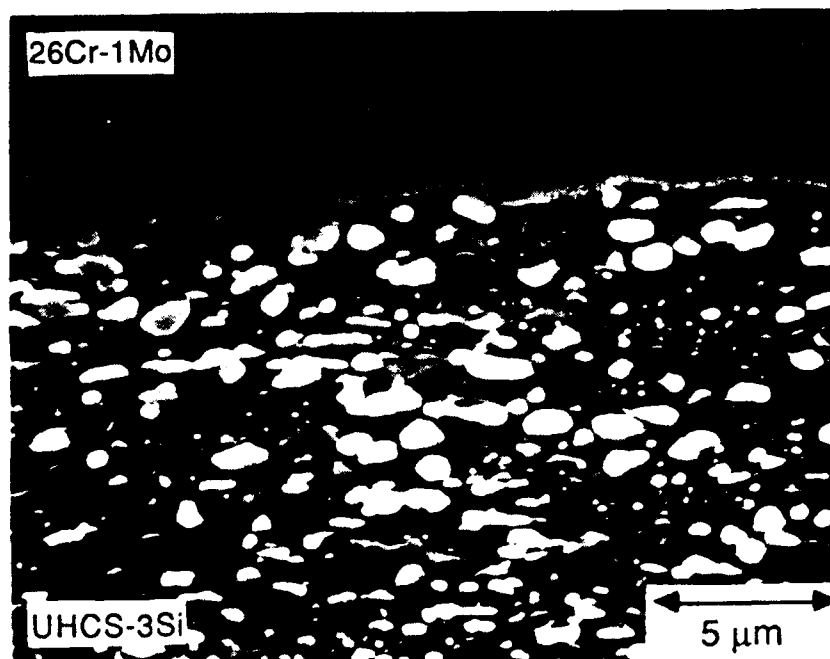


Figure 2. Interface structure in the 26Cr-1Mo / UHCS-3Si laminate after bonding. A ferrite grain size of approximately 2μm exists in the UHCS and there is no evidence of carbon diffusion from the UHCS to the stainless steel.

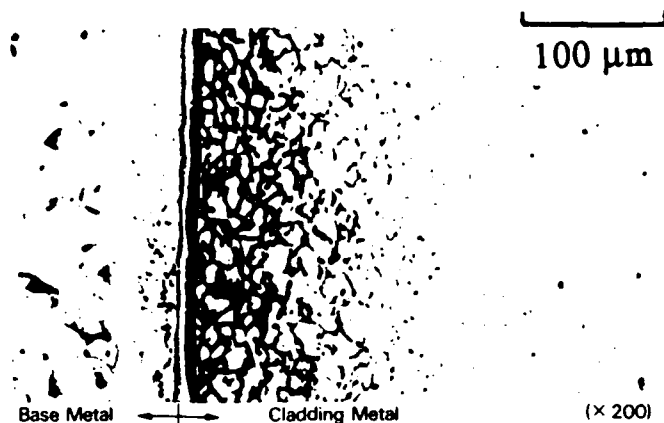


Figure 3. Typical interface structure in commercially produced stainless-clad structural steel. Note the deep penetration of carbides into the stainless steel.

The diffusion of carbon into the stainless steels studied here has not been observed in the laminates as produced in this study. This illustrates one of the potentially useful features of processing stainless clad steels in this way. Using processing routes as described above, an insignificant amount of stainless steel would be degraded by carbon interdiffusion, making possible corrosion resistant stainless clad steels with very thin stainless layers. This would reduce the cost and dependence on strategic resources for such a product.

III. STABILITY OF LAMINATES

The procedure used in this investigation has produced laminates which have very discrete interfaces. Upon heating these laminates near the A_1 temperature for an extended amount of time, however, as is necessary in superplastic forming, carbon will diffuse from the UHC steel into the stainless steel. This is due to the high driving force to form chromium rich carbides. There are two ways in which this interdiffusion can be reduced: by inserting barriers between the UHC and stainless steels, or by altering the thermodynamics of the

system such that the carbides in the stainless steel are not as stable. Practical examples of each approach have been identified.

Several effective barriers have been studied. Copper has a very low solubility for carbon. Thus, thin barriers of copper between stainless and UHC steels have been shown to nearly eliminate the interdiffusion of carbon. Several ferrous materials have also been investigated as barriers. These barriers were selected to inhibit the diffusion of carbon; leading to a significant retardation in interface degradation. The barriers however still maintain a significant solubility for carbon, and, as a result were not as effective as copper.

It has also been shown in static interdiffusion experiments that modifications to the stainless steel composition can slow carbon diffusion into the stainless steel. For example, when a type 304 stainless steel is replaced with type 314, which contains more silicon, a marked reduction in the rate of carbon interdiffusion occurs.

IV. PLASTIC FLOW OF LAMINATES IN ISO STRAIN ORIENTATION

Extensive testing of laminates was performed in the isostrain orientation. The isostrain rule of mixtures,

$$\sigma_c = \sigma_1 V_1 + \sigma_2 V_2$$

has been used to predict the flow stress behavior. As shown in Figure 4, the agreement between experimental data, obtained in strain rate change tests, and predictions based upon this relationship is quite good. In all cases, the agreement was found to be very satisfactory.

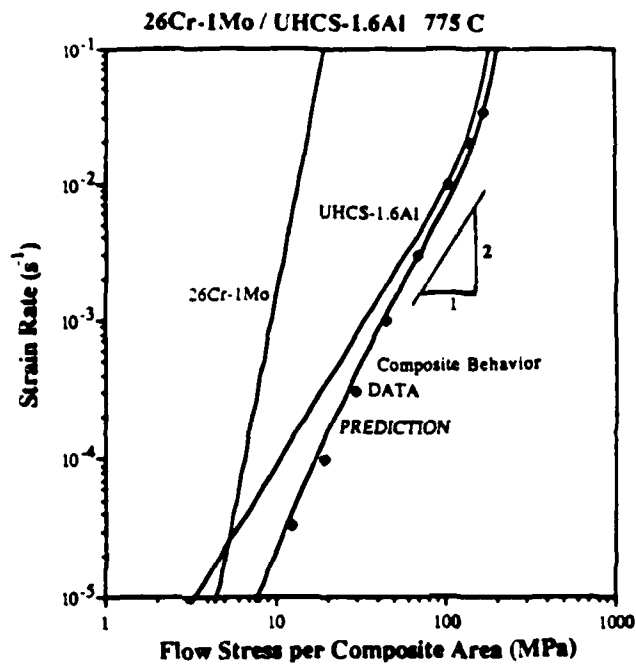


Figure 4. Stress v. strain-rate relationships for the 26Cr-1Mo / UHCS-1.6Al composite. The data for the component materials are presented as volume fraction multiplied by stress. These products are added together at each strain rate to give the predicted composite flow stress as a function of strain rate (solid line). The fit with experimental data (diamonds) is quite good.

The elongation to fracture for laminates of this type can also be predicted through the isostrain relationship. It is well accepted that, under conditions where diffuse necking limits ductility, tensile elongation at fracture is related to the strain-rate-sensitivity-exponent, m . In particular, many empirical and applied mechanics models can be approximated as relating elongation at fracture to the square of m . Utilizing this model and using monolithic UHCS data for normalization, elongation to fracture may be predicted as a function of strain rate. Figure 5 shows such predictions made at three temperatures for the 26Cr-1Mo / UHCS-3Si system (where no diffusion barrier was present), and a comparison is made to experimental data. Although high tensile ductilities are achieved, the experimental data fall well short of the predicted behavior. This is due to the presence of chromium rich carbides formed by interdiffusion, which contributed to a decrease in ductility. When similar predictions and experiments were made for the other systems studied (Table 3), the predictions and data agree quite well, as shown in Figures 6 and 7.

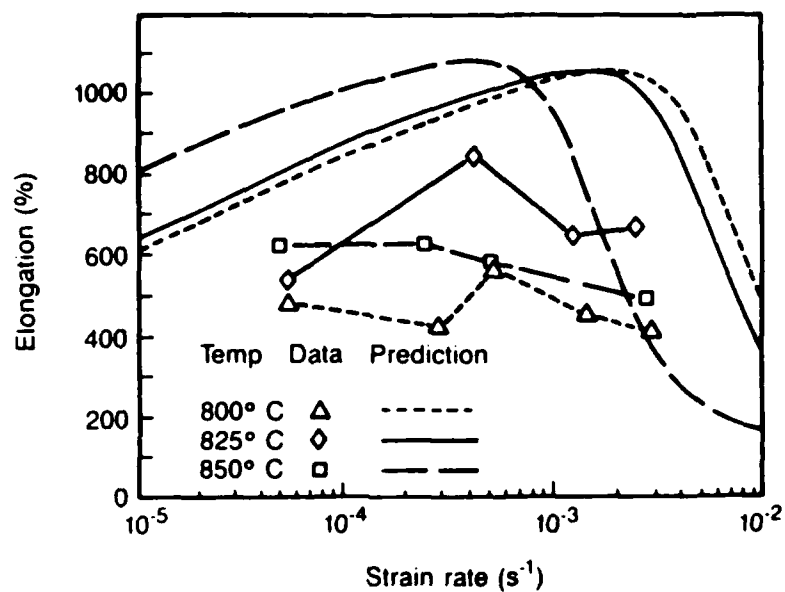


Figure 5. Elongation to fracture v. strain rate for the 26Cr-1Mo / UHCS-3Si laminate. Predictions and experimental data are presented for three temperatures. In this case the experimental elongations to fracture are significantly less than predicted.

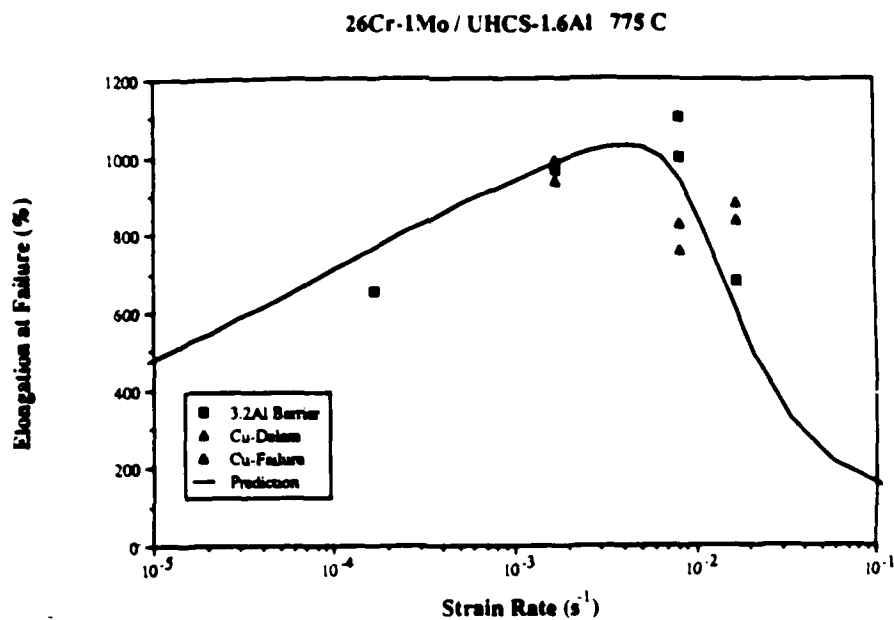


Figure 6. Elongation to fracture v. strain-rate for the 26Cr / UHCS-1.6Al barrier laminates at 775° C. The line shows the predicted behavior, and points show experimental data.

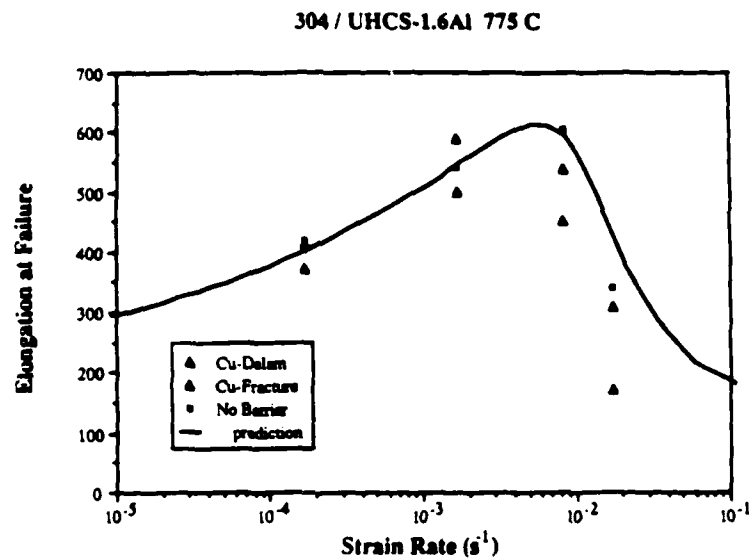


Figure 7. Predicted and experimental elongation to fracture v. strain-rate for the 304 / UHCS-1.6Al laminates at 775°C.

The behavior of the interface in the copper barrier laminates is worthy of note. While these laminated composites typically delaminate (i.e., the stainless steel peels off the UHC steel) near the point of final fracture, they maintain excellent properties along the rest of the interface in the superplastically deformed sample. Figure 8 shows a cross section in the gauge section of the interface of a 26Cr-1Mo / Cu / UHCS-1.6Al laminate strained 300% at 775°C. The interface is perfect. No interdiffusion and no voids are visible. Thus, very discrete interfaces can be maintained even after superplastic forming.

26Cr-1Mo



Figure 8. Interface structure of the 26Cr-1Mo / Cu / UHCS-1.6Al laminated elongated 300% at 775° C. Despite time and strain at elevated temperature, no cavitation or interdiffusion are visible at the interface of this laminated composite.

V. GAS PRESSURE FORMING OF ULTRAHIGH CARBON STEEL AND LAMINATED COMPOSITES

Recently, experiments have begun on the gas pressure forming of monolithic UHC steels and the laminated composites produced in this study. Figure 9 shows the results of forming the 26Cr / IF Iron / UHCS-1.6Al laminate at 775° C with 1.4MPa (200 psi) gas pressure for one hour. The grid on the sample allowed the calculation of the formed thickness distribution in the formed dome. It was found that the thickness was slightly more uniform than predicted by analytical models. This is presumably due to dynamic grain growth induced strain hardening near the pole of the dome.



Figure 9. Example of a gas pressure formed hemispherical section. The 1.96mm thick, 26Cr-1Mo / 1 F Iron / UHCS-1.6Al laminate is the base material. Forming took place at 775° C at 1.4 MPa in one hour.

VI. CONCLUSIONS

Laminated composites based on stainless and UHC steels:

- 1) Can be processed to have very discrete interfaces;
- 2) Have flow stresses which can be predicted by the isostrain rule of mixtures;
- 3) Have predictable tensile ductility, provided deleterious interface phase not form;
- 4) Require barriers for optimal stability;
- 5) Can be superplastically formed with virtually no interface degradation, if appropriate barriers are used; and
- 6) Have useful superplastic forming characteristics.

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